X-Ray Diffractometry

X-ray diffractometry is used to identify specific compounds and phases on the basis of their crystal structures. It is also used to characterize solid-phase transformations and measure crystal lattice parameters. It can be used to measure average crystallite/subgrain sizes and shapes, residual stresses in materials, and determine the microscopic topography of a material.

Principle of Technique

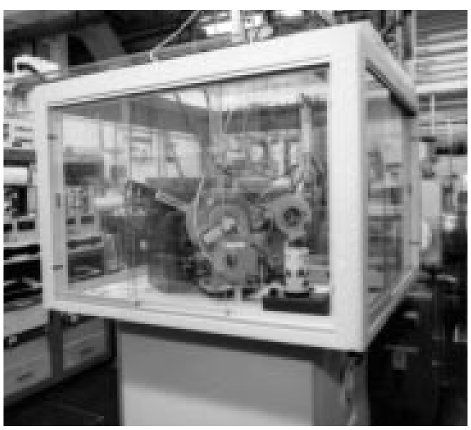
When a randomly oriented aggregate of small crystal fragments (such as a powder) or a single crystal is irradiated with a monochromatic beam of x rays, the various planes of atoms diffract the x-ray beam at angles determined by the spacing between the atomic planes. The diffracted beams can be recorded on a film placed concentrically around the sample. Or the radiation may be detected by a scanning counter and the intensity recorded as a function of scattering angle. Each crystalline phase present in the sample supplies its own unique contribution to the total diffraction pattern. Unknown compounds and phases can be identified by their characteristic patterns. In mixtures, the relative intensities of the scattering patterns can be used to estimate the concentrations of the phases present, and the breadths of the peaks in the pattern are characteristic of the average dimensions of the crystallites present in the sample.

Specific Capabilities

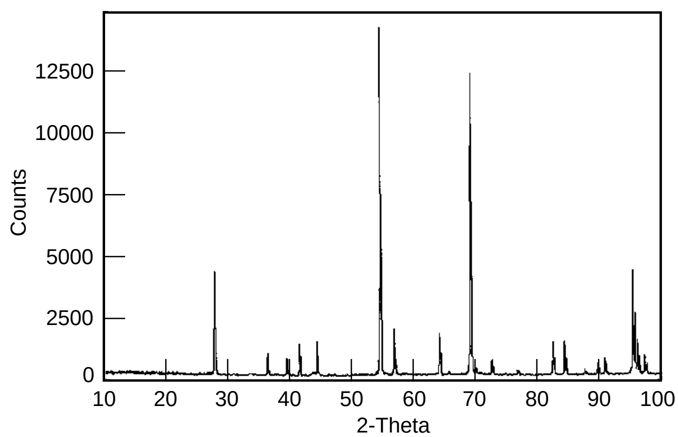
The following table summarizes the characteristics of the equipment and the capabilities in x-ray diffractometry at the Plutonium Analytical Chemistry Laboratory.

Examples of Applications

- Identification and semi-quantitative estimation of the phases present in plutonium metal samples.
- Qualitative determination of the degree of coring in plutonium-gallium alloys.
- Analysis of single crystals for orientation, unit cell size, and location of atoms.
- Production of Debye-Scherrer patterns from single crystals or polycrystalline particles.
- Studies of phase transformations as functions of temperature and pressure.



X-ray diffraction unit in containment enclosure.



This x-ray diffraction pattern of aluminum/titanium nitride shows the presence of rutile crystal structure.

X-ray diffractometry capabilities.

Polycrystalline material analysis			
Sample	Diffractometry	Debye-Scherrer camera	Berg-Berrett camera
Form	Solid, powder	Powder	Powder
Size	1 mg to 1 g	0.1 to 2 mg	mg to several g
Preparation	Some may be required	Some samples need to be Reduced to powder size	Same as for Bebye-Scherrer
Estimated analysis time	Depends on the sample; typically 2 to 3 h	Depends on the sample; typically 5 to 6 h	Few hours
Limitations	Preferred orientation required	Film sometimes hard to read	Film sometimes hard to read
Readout media	Graphics terminal,hardcopy, floppy disk or strip chart	Film	Strip chart or film
Unique feature or capabilities	Inert atmosphere; system automation	Small sample size	Double crystal diffractometry Monochrometric or filtered radiation

Single-crystal analysis				
Sample	Gandolfi camera	Laue camera	Weissenberg camera	Buerger precision camera
Form	Solid	Solid	Solid	Solid
Size	1-µm particle preferred	0.2-mm diam to many cm	0.1 to 0.2 mm preferred	0.1 to 0.2 mm preferred
Preparation	Some preparation is required to bisect sample down to single crystal form	Minimum	Minimum	Minimum
Estimated analysis time	Few hours	Few minutes	Few hours	Few hours
Limitations	Film sometimes hard to read	Film sometimes hard to read	Film sometimes hard to read	Film sometimes hard to read
Readout media	Film	Polaroid or film	Film	Polaroid or film
Unique feature or capabilities	Produces Debye-Sherrer pattern from 1 µm	Very rapid	Can view all of reciprocal space on one film	Undistorted view of single layer of reciprocal space

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